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AN INDUSTRIAL WASTE GUIDE TO THE MILK PROCESSING INDUSTRY

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an Industrial Waste Guide to the

Milk Processing Industry

Prepared by

Subcommittee on Dairy Waste of the

Dairy Industry Committee, in Cooperation

With the National Technical Task

Committee on Industrial Wastes

For the

U.S. DEPARTMENT OF HEALTH, EDUCATION, AND WELFARE

Public Health Service
Division of Water Supply and Pollution Control

Washington 25, D.C.

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Foreword

The control and disposal of wastes is of major concern to many plants in the dairy industry. Optimum reduction and utilization of wastes are essential for economic production in both small and large plant operations. Wastes which cannot be prevented must be disposed of in a suitable manner.

The Nation's water resources are seriously limited in some places. To conserve and protect all water resources for maximum use is mutually beneficial to the individual citizen, our ever-expanding industries, and the Nation as a whole. In recognition of this fact, industries are giving increased consideration to disposing of their wastes in a manner which will not impair the utility of our water supplies for other beneficial uses.

This *Waste Guide to the Milk Processing Industry* is intended primarily to aid management and operators of the dairy industry to reduce, utilize, and suitably dispose of processing wastes. It will also be useful in acquainting consultants and regulatory personnel with the sources and nature of dairy wastes and the progress that has been made in the treatment of such wastes.

The guide was prepared by the Subcommittee on Dairy Waste of the Dairy Industry Committee, which is a trade organization of the dairy industry. It was submitted to the Public Health Service through the dairy industry representatives on the National Technical Task Committee on Industrial Wastes.

The National Technical Task Committee on Industrial Wastes is composed of representatives from the Nation's leading industries concerned with solving difficult industrial waste problems. Its objective is to perform certain technical tasks pertaining to industrial wastes in cooperation with the Public Health Service and others concerned with improving the quality of the Nation's water resources. Preparation of the Milk Processing Guide in the original edition, and this first revision, was one of the tasks assumed by the dairy industry in carrying out this objective. This guide is one of a series being prepared by the National Technical Task Committee in cooperation with the Public Health Service.

Abstract

A plant handling or processing milk and its products may be generally classified as a receiving station, bottling plant, cheese factory, creamery, condensery, dry-milk plant, or ice cream plant.

Dairy plant wastes are made up, for the most part, of various dilutions of whole milk, separated milk, buttermilk, and whey from accidental or intentional spills; drippings allowed to waste by inefficient processing equipment, methods, or operations; washes containing alkali or other chemicals used to remove milk, milk products and partially caramelized materials from cans, bottles, tanks, vats, utensils, pipes, pumps, hot wells, evaporators, coils, churns, and floors; and process washes of butter, cheese, casein, and other products.

Remedial measures may generally be classified as waste prevention, product utilization, and waste treatment. The first two groups include the prevention of avoidable waste of milk solids and the processing and utilization of separated milk, whey, and buttermilk into dry products, casein, albumin, lactose, and stock feed.

The chief disposal and treatment methods in use today are dilution, irrigation, septic tanks, trickling filters, activated sludge, and aeration (modified activated sludge). Dilution or irrigation are usually sufficient where plants are small and isolated or where streamflow is adequate. The trickling filters have been used, averaging 80 to 90 percent reduction in 5-day biochemical oxygen demand. Modified activated sludge is becoming more widely used. Biochemical oxygen demand removals by these processes can reach 80 to 90 percent or more.

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an industrial waste guide to the

Milk Processing Industry

Introduction

This publication represents the combined efforts of dairy waste sanitary engineers, dairy waste technologists, and others of the dairy industry to develop a practical guide for milk plant management and operating personnel.

The subject of milk plant wastes, with methods of recovery and treatment, has been extensively studied for years by State and Federal agencies, experiment stations, sanitary engineers, and other individuals. Many developments have been incorporated in full-size treatment plants for which results under actual operating conditions are available. For the purpose of this guide, such results as are available have been used to demonstrate the various types of treatment.

This publication emphasizes the appreciable reduction of dairy waste which can be accomplished through a waste-prevention program that can be carried out within the processing plant. Practical procedures are described. Some performance data are presented which will serve to emphasize to milk plant supervisory personnel the value of waste prevention methods in reducing total waste treatment costs.

Remedial measures may be classified as by-product recovery, waste saving or waste prevention, and waste treatment.

The section on "Waste Treatment" is not intended to be a comprehensive and complete presentation of dairy waste treatment. Each treatment system has its own special characteristics which may or may not call for special study and modification to bring about satisfactory and efficient performance.

Description of Process

Processes in the milk and dairy products industry may generally be classified as—

a. Receiving.
b. Bottling.
c. Cheese manufacturing.
d. Butter manufacturing.
e. Condensing.
f. Dry milk manufacturing.
g. Ice cream manufacturing.

Since these processes are potential sources of industrial waste in the form of waterborne milk solids in the plant drainage system, the operations involved in each process are given below. In every well-operated plant, waste is reduced to a minimum by proper drainage of equipment and elimination of spillage. However, the wash water used in the regular cleaning of all milk equipment, containers, and plant floors, contains a small amount of milk solids.

Receiving

The milk or cream is received in cans, dumped into a weighing vat, sampled, cooled, and stored in tanks. The milk or cream is then piped to processing departments or loaded into tank cars or tank trucks for shipment to processing plants.

In a growing number of plants, milk is received in tank trucks directly from farms that are equipped with bulk cooling tanks.

Bottling

Processing operations consist of storage, clarifying, filtering, preheating, pasteurizing, cooling, and filling glass, paper, and metal containers with milk, cream, and other products.

Cheese Manufacturing

The raw material is whole milk and, in some cases, cream or separated milk. The raw material is preheated, filtered, pasteurized, and cooled.

Cheese may be classified according to the kind of milk used, the process of making, seasoning, or ripening. Whole milk and separated milk, which may or may not be pasteurized, is run into vats and a "starter"

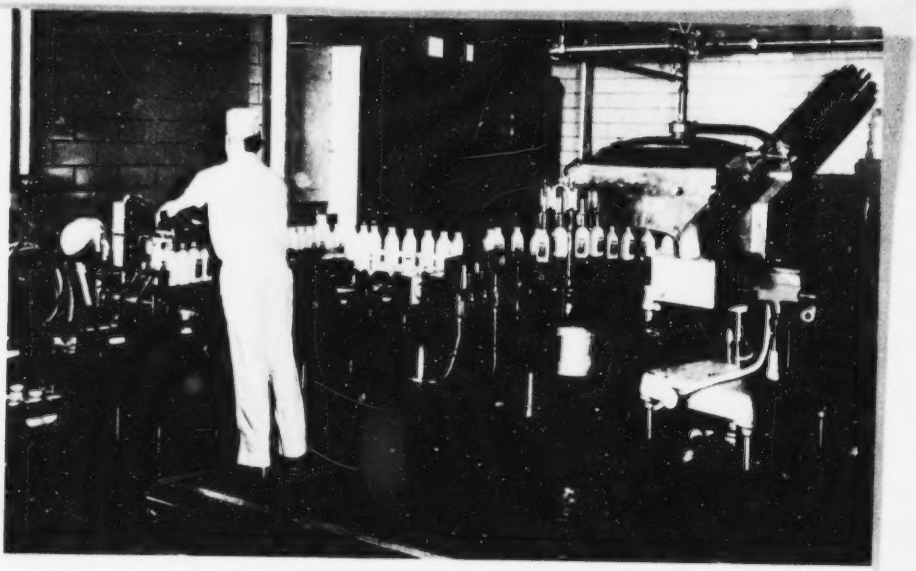

FIGURE 1.—Bottling operations in a modern plant.

(harmless lactic-acid-producing bacteria) is added. The amount of "starter" used runs from 1 to 3 percent of the milk weight. The mixture is allowed to set and ripen at controlled temperatures until proper acidity has developed. Rennet may be added to cause a curd to form and facilitate its separation from the whey. Prior to this separation, the mixture is allowed to set and ripen at controlled temperatures until the required acidity is developed. The casein in the curd may be peptonized, after separation from the whey, and through proper storage the characteristic flavors developed. For process cheese the ripened product is ground, melted, pasteurized to stop ripening, and packed while hot.

Butter Manufacturing

Whole milk or cream is processed into butter and other products. When whole milk is received, it is separated centrifugally into the cream fraction and the separated milk fraction. The separated milk may be processed into other dairy products for human consumption or for animal feed.

In the manufacture of butter, cream is pasteurized and may be ripened with a selected acid and flavor-producing culture in preparation for the churning. In the churning process, agitation of cream at low temperature, generally between 45° and 55° F., changes the type of emulsion from a fat-in-water phase to a water-in-fat phase. As this is done, butter granules

FIGURE 2.—Cheese vats equipped with automatic agitators.

FIGURE 3.—Bulk "starter" room in cheese manufacturing plant.

of a visible size gather and trap small droplets of water in the fat. The butter is agitated in the buttermilk until such time as the particles of butter are of the size of small peas. At this time, the buttermilk is drained from the churn. The butter is washed, standardized for moisture and salt, "worked" until it becomes a uniform mass, and then withdrawn from the churn for packaging and distribution. The buttermilk is collected and diverted to animal feeding or is processed by condensing or drying.

Condensing

Whole milk, separated milk, or other dairy products are evaporated to produce a concentrated product. Unsweetened evaporated milk is the product of this group in the largest production.

For the production of unsweetened evaporated milk, the whole milk is first preheated and then evaporated by a method in which the milk flow through the evaporator is carefully regulated to produce a continuous discharge of product having the desired concentration.

The evaporator consists of a closed system equipped with suitable heating surface. The source of heat is generally steam. A vacuum sufficient to cause the milk to boil at greatly reduced temperatures is maintained in the system. Vapors are removed through a top outlet and condensed by water or other cooling media.

After evaporation, the milk is homogenized; i.e., forced at high pressure through a closely set valve to break up fat globules and produce a fine suspension. Cans are filled and sealed by machine, after which they are sterilized.

The sterilizing may be done in either a continuous or batch type of operation. The result is the same with either type, in that the cans are fed into a retort of suitable size and construction, and are subjected to a temperature of 235° F. or more for a period of 15 to 25 minutes; they are then cooled by circulated water and removed. When dry, the cans are stored or prepared for shipment to market.

Sweetened condensed milk is produced in a similar manner, except that sugar is added. This product is evaporated to a higher concentration. Sterilizing after canning is not necessary since the sugar acts as a preservative.

Other condensery products such as condensed nonfat milk, whey, and buttermilk are manufactured by variations of the above processes.

Dry Milk Manufacturing

Dry milk products, including dry whole milk, nonfat dry milk and dry buttermilk solids may be made from the corresponding fluid product by one of the following processes:

(1) Spray process: Preheated condensed milk is sprayed either centrifugally or under pressure through an orifice into a chamber through which a current of heated air is passed. The finely atomized droplets dry almost instantly and are removed by various collecting systems.

(2) Roller process: The milk or condensed milk is fed into a trough formed between two steam-heated drums revolving in opposite directions. The regulated clearance between the drums produces a thin film of milk on each drum which dries quickly. The dry film is scraped off by a fixed knife and pulverized.

Ice Cream Manufacturing

Ice cream mixes are made from various formulas by combining cream, milk, and condensed milk or dried milk products with flavoring, sugar, and other ingredients. The mix is then pasteurized, homogenized, and cooled. In some cases the fluid products are blended and then concentrated in an evaporator. After cooling, the mix is ready for storage or freezing.

The ice cream mix is partially frozen to a semisolid mass in either a batch or continuous freezer. It is then packaged and the freezing is completed by storage in a subzero hardening room or by other quick freezing methods.

Raw Materials and Products

The approximate composition of some common dairy products are shown in table MK–1. Ratios of products to raw material are shown in table MK–2.

TABLE MK-1.—*Approximate average composition and Biochemical Oxygen Demand (B.O.D.) of some common dairy products* [1]

Dairy product	Butterfat (percent)	Protein (casein and lactalbumin) (percent)	Milk sugar (lactose) (percent)	Added sugar (percent)	Ash (percent)	Acidity as lactic (percent)	Total solids (percent)	Organic solids (percent)	B.O.D.[2] (percent)
Whole milk [3]	3.9	3.2	5.1	0	0.7	.....	12.9	12.2	10.3
Evaporated milk	7.9	6.7	10.1	0	1.4	.....	26.1	24.7	20.8
Separated milk	.1	3.3	5.3	0	.8	.....	9.5	8.7	7.2
Nonfat dry milk solids	.9	36.9	50.5	0	8.1	.....	96.4	88.3	73.7
Sweetened condensed separated milk	.3	10.4	16.8	40	2.5	.....	70.0	67.5	50.2
Buttermilk [4]	.5	3.4	4.3	0	.7	0.6	9.5	8.8	7.2
Whey [5]	.3	.9	4.9	0	.6	.2	6.9	6.3	3.5
Cheddar cheese	35.5	26.6	1.5	0	3.5	.....	67.1	63.6	60.0
Cream	40.0	2.2	3.0	0	.4	.....	45.6	45.2	39.9
Ice cream mix	12.0	3.9	5.9	15	8	.....	37.6	36.8	29.2

[1] Averages from various sources.
[2] Percent B.O.D. = number of pounds of oxygen used by 100 pounds of product in 5 days at 20° C.
[3] Since the composition of whole milk varies considerably because of many factors, the values or individual samples may show considerable difference.
[4] Ripened cream buttermilk.
[5] American cheese whey.

TABLE MK-2. *Ratio of products to raw material*

Process	Raw material	Products (pounds per pound raw material)
Separation	Whole milk (4 percent butterfat)	Cream, 0.12 (30 to 40 percent butterfat). Separated milk, 0.88.
Cheese making	Whole milk	Cheese, 0.10. Byproduct cream, 0.02. Whey, 0.88.
Do	Skim milk	Cottage cheese, 0.16. Whey, 0.84.
Butter churning	Cream (30 percent butterfat)	Butter, 0.37. Buttermilk, 0.63.
Do	Butterfat	Butter, 1.25.
Milk drying	Whole milk	Dry whole milk, 0.12.
Do	Nonfat milk	Nonfat dry milk solids, 0.086.
Do	Sweet cream buttermilk	Dry buttermilk solids, 0.093.

Sources and Volume of Waste

Sources of Industrial Waste

The industrial waste from dairy plants consists of the waterborne milk solids which enter the drainage system. They are classified according to their source as follows:

1. Washings and rinsings from cans, equipment, and floors.
2. Spillage, freeze-on, overflow, and leakage due to worn-out equipment, and improper operation.
3. Entrainment from evaporators.
4. Byproducts, such as buttermilk, separated milk, and whey which have not been utilized.
5. Spoiled or damaged raw or manufactured products or byproducts.

Sources of Domestic Waste

The domestic waste originates in lavatories, locker rooms, and laundries. Its volume is controlled by the number of employees.

Volume of Industrial Waste

The volume of wastes discharged from a dairy plant processing any particular material may vary widely, depending mainly upon the availability of water and the control of all water-using operations within the plant. This is particularly true of milk-products plants where rinse and wash waters of continuous flow operations are not necessarily proportional to the volume of production, where cooling and condensing waters may or may not be recirculated in evaporative cooling equipment, and where the excess of byproducts such as skim milk, buttermilk, etc., may be accidentally discharged to the sewers.

Clean water from coolers, ice machines, and other equipment, and roof drainage are not considered as wastes. This water adds volume to the waste unless it is segregated through separate drains, and may have an adverse effect on treatment.

Strength of Wastes

The most common processes in the dairy industry and the pounds of B.Q.D. which can be expected in the material lost to the sewer in each process with the use of reasonably modern equipment and careful operation are indicated in table MK–3.

Because of the wide variation in individual cases, these figures should not be relied upon in the design of waste treatment systems. They are given to serve as a guide in reducing losses within a given process which show wide variation from the average. These figures are representative of ordinary wash wastes and process losses, and do not include byproducts, separated milk, buttermilk, or whey except the amounts included in normal cheese and butter washes.

Further reductions of waste below the figures indicated can be accomplished by proper waste-saving practices and under carefully maintained and controlled conditions.

Effects of Pollution

The varying ability of streams to tolerate dairy waste depends largely upon the actual volume of water in the stream as related to the volume of waste discharged at a given time and the rate of flow of the stream. Milk wastes are organic in nature and the pollution effects are almost entirely due to the oxygen demand (oxygen actually removed) which such wastes impose on the receiving stream. Fresh wastes may be acid or alkaline. When receiving waters cannot provide sufficient oxygen to maintain aerobic decomposition, the stream will not support fishlife. If this condition continues, anaerobic decomposition occurs and offensive black sludge is formed. The heavy black sludge deposits and strong pigpen odors caused by the putrefying casein are characteristic of milk waste pollution.

TABLE MK-3.—*Approximate quantities of B.O.D. in various process wastes* [1]

Process	Pounds of 5-day B.O.D. per 10,000 pounds milk or milk equivalent [2]
Receiving and cooling milk	4
Tank-truck delivery to and from plant including washing tank truck	1
Storage of fluid milk, separated milk or whey in tanks	.5
Storage of other fluid products	1
Evaporating whole milk, floor waste	2
Evaporating whole milk, entrainment loss	1
Canning and sterilizing evaporated milk	2
Spray drying whole milk	1
Cream separating	2
Cream pasteurization, cooling and can filling	2
Separated milk pasteurization, cooling, and can filling	2
Cottage cheese or casein manufacture	16
American-cheese making, unwashed curd	10
American-cheese making, washed curd	16
Separated-milk condensing, plain, floor waste	3
Separated-milk condensing, plain, entrainment loss	.5
Separated-milk condensing, sweetened, including barreling floor waste	6
Separated-milk condensing, sweetened, entrainment loss	.5
Separated-milk condensing, superheated	12
Separated-milk drying, spray process	1
Separated-milk drying, roll process	16
Whey condensing, sweet, floor waste	8
Whey condensing, sweet, entrainment loss and volatile	2
Whey condensing, acid, floor waste	8
Whey condensing, acid entrainment loss and volatile	4
Whey drying, spray	5
Buttermilk condensing, floor waste	8
Buttermilk condensing, entrainment loss and volatile	4
Milk pasteurization, cooling and bottling (glass)	8
Milk pasteurization, cooling and bottling (paper)	6
Ice-cream-mix making, vat	4
Ice-cream-mix making, pan	4
Ice cream freezing	.5
Cultured-buttermilk making	5
Butter churning and washing	2

[1] Table adapted from "Waste Prevention in the Dairy Industry."
[2] "Milk equivalent" is the quantity of milk product or byproduct from 1 pound of milk.

Waste Prevention

The prevention or elimination of the waste of milk fats and solids is an economic necessity. Such losses are *costly* because of the actual product loss, and because of the amount of money necessary for waste treatment equipment and its operation. Excessive waste originates from the same sources in all dairy-processing operations. Management and those in charge of operations will find it profitable to study this problem and take appropriate action to prevent excessive waste. The following discussion deals with the more common origins and causes of such losses and their suggested means of correction.

Leakage and Drippage

The constant and continual loss of milk from improperly assembled or fitted sanitary lines, pumps, plate and tubular heaters and coolers, bottle fillers, and valves and connections on all dairy equipment is not uncommon. The volume and cost of such losses are much higher than is generally recognized.

Leakage may occur at any piece of equipment where two component parts must be assembled either with or without a gasket; for example, sanitary fittings, pumps, homogenizers, plate pasteurizers, etc. It is essential that proper gaskets be used, and where per-

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manent-type gaskets or ground joints are in use, that they be properly maintained. Filling machines for cans, glass, and paper bottles require care and attention. The valves must be properly assembled and kept in good condition to prevent waste.

Since repair of fittings and pumps is not usually possible during operations, sanitary lines and pumps should be mounted high enough, when convenient, to allow placing of drip-collecting pans under them when leakage occurs.

Air leakage in pumps causes foaming. This foam overflows and carries with it considerable milk solids. Such leaks may occur on the suction line or through rotary seals or packing.

Overflow and Spillage

Loss of milk may occur because of overflow of cans, containers, vats, tanks, etc. This can be greatly reduced, if not completely eliminated, by using the proper equipment or adopting the necessary measures to control the situation. Receiving tanks used in conjunction with weigh cans in the receiving room have a limited capacity, and the 3–A Sanitary Standards definitely limit such maximum capacity. Because of this and the rapidity with which milk is received, alarms and controls should be installed to prevent overflow.

The safest, simplest, and most economical way to prevent overflow from receiving tanks is to provide a suitable type of pump for removing the milk. This pump should be kept in continuous operation when milk is being received and its capacity must be such that it can remove the milk as fast as it is delivered.

Storage and holding tanks and pasteurizing and cheese vats must be watched carefully or equipped with mechanical means to prevent overflow. Sanitary liquid level control devices to start and stop pumps, and alarms which will notify the operator when the tank is nearly full, are recommended. The use of C.I.P. (cleaned-in-place) equipment and pipelines is desirable as these installations materially reduce the amount of leakage.

Spillage of milk is recognized to be due largely to careless handling by plant personnel. This can best be prevented by developing a dairy waste prevention program from which plant employees will realize the economics involved.

Freeze-On

The freezing-on of milk and milk products on coolers and freezers may be an important source of milk solids loss. Proper design of equipment with adequate refrigerant controls and proper operation will reduce these losses to a minimum. Coolers should be designed with proper controls so that the milk can be cooled to the desired temperature by a refrigerant whose temperature is not lower than 32° F.

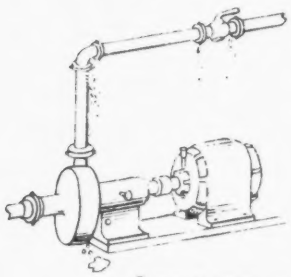
Willful Waste

When a large volume of milk solids enters the sewerage system from one plant, it is probably done willfully in that no effort was made to keep such solids out of the sewer. Such action may cause an endless amount of trouble. If the discharge goes to a stream, it may so impede natural purification processes that the stream will become a nuisance; if discharged to a treatment system, it may disturb the normal bacterial activity of the system to such an extent as to put it out of commission. Refer to the section on "Utilization of Dairy By-products." Large volumes of milk solids should not be discharged directly to the sewer.

Residual Waste

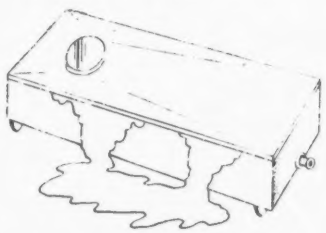
Residual waste is the milk or milk product which adheres to the surface of cans, tanks, and processing equipment. The total losses may reach amazing proportions unless care is taken to be sure that as much as possible of these residuals is kept out of the sewer.

LEAKAGE & DRIPPAGE



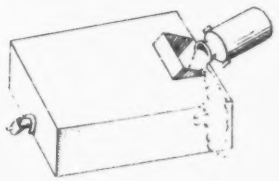
Pump & Fittings

OVERFLOW



Receiving Tank

SPILLAGE



Careless Handling

Milk Receiving: Losses in the receiving room can be materially reduced by proper dumping of cans and providing adequate time for drainage from the individual cans. In plants where such precautions are not taken, it has been observed that the losses may exceed 100 pounds for every 25,000 pounds of milk received. The best way to accomplish this with a straightaway washer is the use of the Western Dump. This allows a few seconds for the drainage of milk from the can and reduces losses approximately 50 percent.

Milk continues to drain from the can after it is moved forward from the weigh can hopper. By means of an extension with a drip-collecting pan at the entrance of the can washer, additional drain space is obtained and a large part of the residual milk may be recovered. Additional milk can also be recovered by means of a volumetric spray nozzle which sprays the interior of each can with 3 to 4 ounces of water. These washings can be collected and used for animal feed. Such methods are recommended to materially reduce the amount of milk solids going to the sewer.

For handling cream cans, steam jets with can-operated valves can be placed in the can-washer extension. The cans are emptied into the strainer hopper or forewarmer, but they are steamed to remove the residual cream and butterfat as they pass over the steam jets in the extension attached to the can washer.

For handling fresh milk, at least two drain positions are required and at least four positions are required for cream and concentrated milk products.

Milk Processing: The drainage of dairy products from sanitary piping, evaporators, storage tanks, pasteurizers, and other receptacles adds a considerable amount of milk solids to the waste. This is especially true where condensed and dry milk products are concerned.

Sanitary lines should be pitched to accomplish complete drainage. Tanks, vats, and similar equipment should be designed to permit maximum drainage. This drainage should be collected in sanitary containers and either processed or used for animal feed.

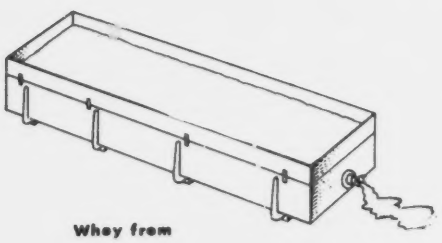
Prevention of waste losses may be further effected by spraying a small quantity of water on the inside walls of equipment and similarly rinsing sanitary lines and collecting such rinsings so that they will not pass to the sewer.

Butter Processing: Churns and butter printers should be so conditioned that butter will not readily adhere to the surface. All butter should be removed before cleaning.

Ice Cream Processing: Ice cream and ice cream mixes are heavier in body than other milk products and unless care is taken to allow time for complete drainage from equipment, or a small quantity of water is used for rinsing, the losses to the sewer may be considerable.

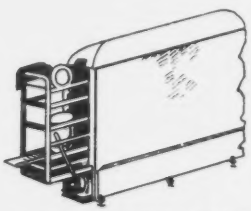
Dry Milk Products Processing: Milk and nonfat milk products are dried by the spray or roller process. That which is dried in spray chambers should be so confined that virtually complete collection is possible. The air discharge from spray driers should be passed through collection equipment. Cyclone separators and bag-filling rooms should be checked at regular intervals for satisfactory operation. Losses from the roller driers may be due to the free product which may accumulate on the floors and leakage from end dams on the rollers. All dry milk products should be collected by sweeping and vacuum cleaning prior to washing operations. A dairy waste prevention program should include adequate facilities for collecting all dry milk solids. This cannot be too greatly emphasized because a very small amount of milk solids (in dry form) will greatly increase the strength of plant waste.

WILLFUL WASTE



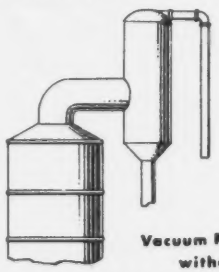
Whey from Cheese Vat

RESIDUAL WASTE



Cans Inadequately Drained before Washing

CARRY-OVER



Vacuum Pan without Entrainment Separator

Cream Processing: Separators of the open type may produce considerable quantities of foam. Foam carries considerable milk solids. The closed, or airtight, separator is recommended. In this equipment it is important that gaskets be maintained in good condition.

Cheese Processing: This operation can be a major source of buildup of milk solids going to the sewer. Whey and cheese washings have a high B.O.D., and if allowed to escape to the drain will materially increase the pollution and require increased costs for waste treatment. To avoid spillage during mixing, cheese vats should not be filled higher than to within 3 inches of the top rim. Ample capacity for handling whey should be provided. Whey is customarily discharged from the cheese vats into shallow, open pans from which it is pumped to storage. The flow of the whey is regulated manually, and if the capacity of the pump is exceeded, or the pump becomes airbound, the whey runs down the drain. Two improved methods of whey removal are outlined in the following paragraphs:

A direct connection through a three-way valve may be made from the vat to the pump suction line. A removal screen is located in the vat over the end of the suction line to separate the cheese particles from the whey as it is removed.

In the second method, a small tank is mounted at the end of the cheese vat so that the top of the tank is at the same level as the top of the vat. A connection is provided from the vat to the tank so that the whey enters the tank through a strainer. The whey is pumped directly from this tank. The strainer is removable so that the cheese particles which are retained can be returned to the vat.

The pump used in both of these methods, when in a fixed position, should be of the positive type; when pumps of the portable type are used, centrifugal pumps are satisfactory. Cheese hoops should be filled in the vat. Suitable containers should be located under each press to prevent press liquor from entering the sewer. Cheese curds spilled on the floor should be swept up and not washed to the floor drains.

Carryover

Milk and its products are concentrated in single- or multiple-effect evaporators. If this equipment is incorrectly designed or improperly operated, large amounts of product may be discharged with the cooling water and condensate. Usually a high dilution of the product in condenser water prevents such losses being readily recognized while, at the same time, it is adding materially to stream pollution. Where entrainment losses are experienced, it is recommended that level controls and entrainment separators be installed.

Suggestions for Effective Waste Prevention

1. Adopt and maintain a definite waste prevention program. Waste prevention committees and group discussions will help make employees "Waste Prevention Conscious."
2. Instruct plant personnel in the proper operation and handling of equipment.
3. Make a thorough study of plant operation to determine where losses occur.
4. Provide ample equipment, especially for receiving, cooling, and storing to take care of maximum volumes so that there can be no spoilage due to delays in handling.
5. Mark all valves clearly, especially multiport, so that it is practically impossible for inexperienced help to turn the valve the wrong way.
6. Handle with extreme care all sanitary fittings, valves, rotary seals, and pump parts during every phase of operation to prevent marring which may cause leaks.
7. Provide reliable standby power so that processing can be completed as quickly as possible in case of a breakdown of the regular source of power.
8. Provide accurate temperature controls on plate, internal tubular, and surface coolers to prevent freezing-on.
9. Eliminate valves on the outlet side of internal tubular or plate heaters or coolers to avoid waste due to blown gaskets.
10. Install suitable liquid level controls with automatic pump stops, alarms, or other devices at all points where overflows are likely to occur.
11. Vats and tanks should have well-rounded corners, be properly pitched, and be installed high enough from the floor for easy draining and rinsing into standard buckets.
12. Install equipment such as vats, tanks, and processing machinery designed to reduce to a minimum losses due to leaky joints, gaskets, packing glands, and rotary seals, etc.
13. Install all sanitary lines so that they can be drained thoroughly and prerinsed with a small amount of water into standard buckets. This residual material is suitable for animal feed.
14. Give distinctive markings to the different types of fittings on plate type heat exchangers to avoid milk being pumped into the water side of the exchanger.
15. Provide and use proper splash shields on surface coolers.

16. Never fill cheese vats, ice cream mix vats, pasteurizers, or cooling tanks to such a high level that spillage will occur when the product is agitated.

17. Worn-out or obsolete equipment or parts of equipment, including sanitary valves, fittings, and pumps, should be repaired or replaced. When a leak cannot be repaired during processing, buckets should be provided so that the product is not allowed to go down the floor drain.

18. Foam contains a considerable amount of milk solids and should be kept out of the sewer. Common sources of excessive foaming are open-type separators, splashing when filling tanks, air sucked in through leaky connections in lines under partial vacuum, through leaky packing, and through faulty rotary seals or pumps.

19. Replace or repair leaky milk cans.

20. Provide the best possible conveyor levels and unloading facilities to avoid cans being upset during unloading, and maintain conveyors so that cans will not tip over.

21. Where permitted, use grid style (Western Style) dumping of milk cans. Where practical, eliminate the use of milk cans and substitute tank truck pickup on farms and between plants.

22. When unloading tank trucks, avoid spillage from the sanitary pipe or hose connections. Be certain the tank is empty before disconnecting the sanitary pipe or hose.

23. Provide an adequate drip saver between can dumping and can washing. A product saving prerinse (2 to 4 ounces of water per can) should be used at the entrance to the can washer over the drip saver pan extending for 2-3 drain positions. Such prerinsings can be used for animal feed.

24. Do not use a constantly running water hose in any room. Eliminate the cause of spillage, rather than just wash it away after it has occurred.

25. Avoid the use of hot wells for preheating because of the danger of filling them too full or of their boiling over. Use regular plate, tubular, or surface heaters. If hot wells must be used, equip them with liquid level controls which operate alarms or pump stops with good temperature regulators.

26. Operate evaporators at low liquid level to prevent boiling over.

27. Design all new external tube-chest evaporators with a tangential inlet from the tube chest to the evaporating space. Equip all coil or calandria evaporators with efficient entrainment separators. So-called splash disks in the top of the evaporators will not do the job unless carefully operated under the rated capacity with a low liquid level and at relatively low vapor velocities.

28. Wherever possible, equip the condensers of evaporators with a full barometric leg so as to eliminate the possibility of spoiling products by sucking water back through the condenser into the evaporator in case of pump or power failure. A full barometric leg will be very helpful in maintaining a steady vacuum which again helps to reduce entrainment losses.

29. In filling or moving cans or barrels of liquid dairy products, take extreme care to avoid spillage.

30. Where sugar is used in the manufacture of dairy products, take care that it is not spilled on the floor and washed into the sewer. Sugar will pollute the stream just as much as will milk solids.

31. When condensed products have been removed from vacuum pans into storage or cooling vats, utmost care should be taken that no cleaning solutions be allowed to enter the vats, thereby spoiling the product.

32. All dust from spilled or wasted products in milk-drying operations should be scraped or swept up with the aid of a vacuum sweeper. These products should be collected and disposed of for animal feed. Water from hoses should never be allowed to flow constantly over powder room floors.

33. Provide a foolproof whey collection system and avoid leaks from valves and fittings on whey lines which will result in ultimate loss of whey to the sewer and in floor corrosion.

34. Provide standby pumps for pumping whey from cheese vats to storage tanks in case of power or pump failure.

35. Provide whey storage tanks of twice the maximum daily volume for all types of cheese and casein plants.

36. Sweep up all spilled cheese curd particles from the floor. Do not wash them down the sewers.

37. In casein operations, collect all casein dust and collect all fine particles with proper settling equipment or filter presses. This can be used for animal feed.

38. In lactose manufacturing plants, evaporate lactose mother liquors and lactose wash liquids and use them for animal feed. Storage and catch basins must be available. Remove albumin or other cake carefully from the filter presses so that none is lost to the sewer.

Problem of Utilization of Dairy Byproducts

The most important factor in waste saving and waste disposal is the complete utilization of all milk solids so that none will be discharged to the sewer. Table MK–1 indicates the organic solids content and the pollution effect in terms of B.O.D. equivalent.

Several important and practical difficulties confront the dairy industry. The production is highly seasonal and small plants cannot afford a large investment in equipment which will be used for a limited period during the year. Milk product prices are influenced to a marked degree by the competitive prices of other human and animal feed products. It is difficult to determine a few years in advance which product is going to be the most profitable outlet for milk solids. The ideal dairy plant should be able to make any and all kinds of products so that it may realize a satisfactory profit during any given period.

The principal products that are usually in excess during the season of high production are indicated below together with the possible uses for each:

Separated milk: For plain condensed separated milk, sweetened condensed separated milk, nonfat dry milk, casein, paper coatings, paints, plastics, and adhesives.

Buttermilk: For concentrated buttermilk and dry buttermilk solids.

Whey: For pasteurized sweet whey, whey protein, dry whey, condensed whey, sweetened condensed whey, lactose, riboflavin, ethyl alcohol, lactic acid, protein hydrolysates, whey cheese (primost or ricotta), and vinegar.

Other byproducts: The processing of some of the byproducts listed above produce additional byproducts for which uses must be found to avoid excessive wastes. Such residual products are serious factors in stream pollution.

Waste Treatment and Disposal

At locations where waste prevention and byproduct recovery have been shown to be insufficient to correct or reduce stream pollution from dairy waste, a change in the method of disposal or the installation and operation of waste treatment facilities will be necessary. Frequently, increased capital expenditures for waste prevention and byproduct recovery equipment are justified by the resulting reduction in the cost of waste treatment facilities.

The cost of separate waste treatment facilities will vary considerably because of climatic conditions, type of treatment, and many other local and influencing factors. The cost may range from $150 to $2,000 for each pound of B.O.D. per day in the waste. This wide range results from the fact that the cost figures cover not only ideal conditions where minimum expenditures are required but also those instances where difficult construction conditions are encountered, or where rather extensive collection or outfall sewers are needed. Irrigation systems are much less expensive, as indicated in a subsequent section.

The possibility of discharging the residual waste into a municipal sewage treatment plant should be thoroughly investigated before embarking upon the design of separate waste treatment facilities. It is important that dairy management discuss and reach agreements with both State water pollution control authorities and appropriate municipal sewage treatment officials before making a final decision pertaining to the treatment and disposal of waste. The disposal of wastes into a municipal sewerage system may prove more satisfactory and economical than separate treatment.

Several methods of treating dairy waste have been investigated and developed. At the present time, the most satisfactory means are aeration (modified activated sludge), irrigation, biological filters, and activated sludge. Aeration- and irrigation-type plants do not (in most installations) require as large a capital investment as do biological filters and activated sludge systems. However, the operation costs of aeration plants are generally higher because of power requirements for supplying air. A brief discussion of each of these methods follows. Information on other treatment methods, and additional information on the methods presented herein, may be secured from the technical literature listed in the bibliography.

Careful technical and scientific consideration is required to obtain satisfactory results in the treatment

FIGURE 4.—Disposal of wastes on land by tank truck.

of dairy waste. In designing any treatment system many important factors must be considered, the most important of which are the following.

1. Sound and dependable data must be collected, including the volume and strength of the waste as related to the pounds of milk intake and the variations in manufacture and processing. Seasonal variations in milk volume and in processing warrant particular attention in developing design data.

2. Variations in the tolerance of the receiving stream in its ability to accept the effluent should be determined to insure compliance with State stream sanitary laws.

Overloading and underdesign of treatment systems are two of the most serious causes of unsatisfactory operation. From these causes may stem many treatment problems: odor nuisances, ponding of trickling filters, bulking activated sludge, and poor quality effluents, with resulting deterioration in the quality of the receiving waters.

Aeration (Modified Activated-Sludge-Type Treatment)

A number of dairy waste treatment plants are now operating on aeration principles established by the initial studies of H. A. Trebler, confirmed on a laboratory scale by S. H. Hoover, N. Porges, and others of the Eastern Regional Research Laboratory, USDA, in Philadelphia, together with data from a full-scale pilot plant under R. R. Kountz at Pennsylvania State University.

These plants differ from activated sludge plants in that the sludge developed is aerated until stabilized, whereas in the activated sludge process, the sludge is removed to digestion units for stabilization or is disposed of otherwise. Extended aeration at a high rate results in the destruction of the cells by their own metabolism (endogenous oxidation), thus reducing the sludge volume as well as stabilizing it.

A continuous flow system consists of (1) an equalization tank (which may or may not be used, but which is to be preferred) into which the raw waste flows and

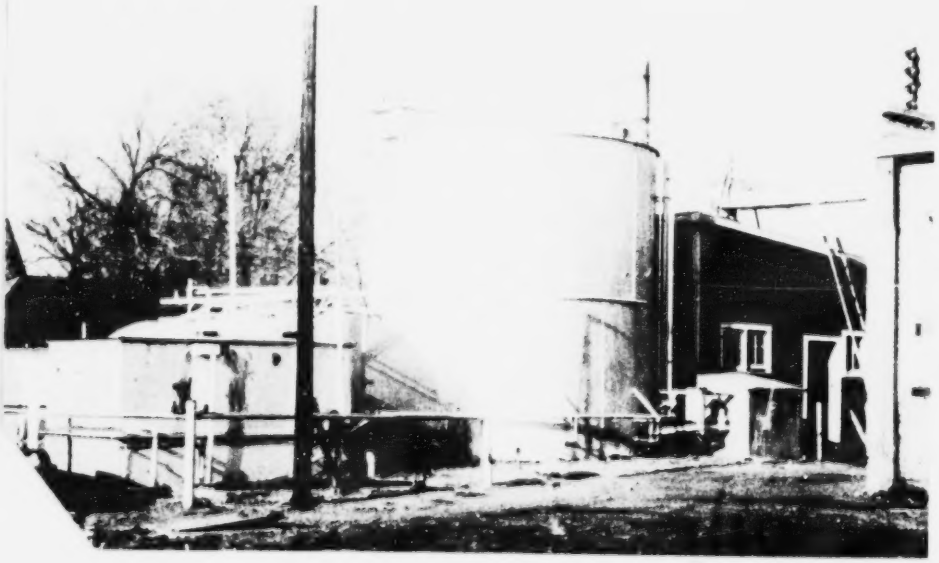


FIGURE 5.—Steel fabricated waste treatment system installed at milk plant.

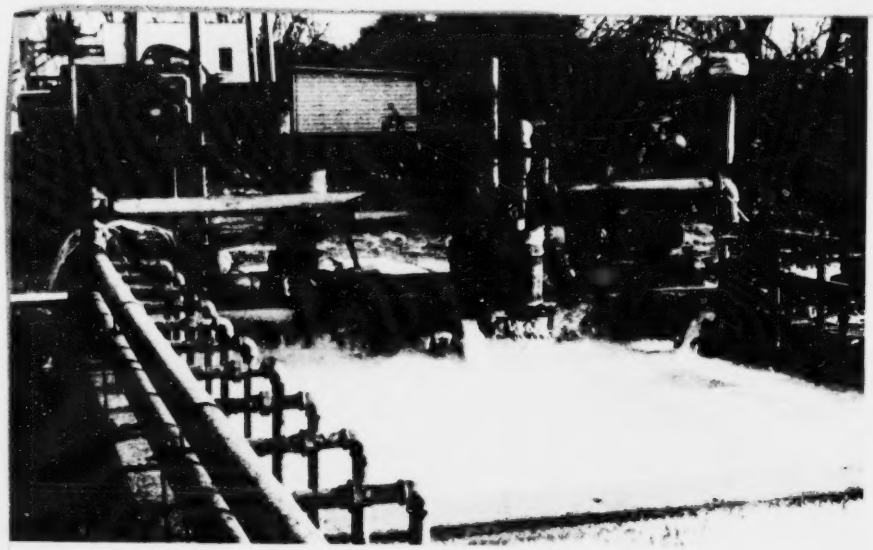


FIGURE 6.—Aeration facilities at milk waste treatment plant.

the volume and strength of the waste are equalized before the waste is pumped to the aeration tank; (2) the aeration tank where the waste is vigorously agitated and oxidized by means of air introduced through diffusers, or other devices, to convert the organic matter to cell material and final oxidation; (3) the settling tank which receives the mixed aerated liquor and in which the suspended solids are removed for return to the aeration tank. The clear supernatant liquid flows to the stream.

In the Fill-and-Draw Aeration system, the cell mass, along with the organic matter, is allowed to settle. After assimilative aeration during the night and a minimum of 2 hours' settling time, the clarified liquor is withdrawn as effluent. The retained sludge (3,000 to 15,000 p.p.m. suspended solids) is then aerated to satisfy oxygen requirements for stabilization and serves as seed for the next waste load. Complete oxidation requires 1.25 pounds of oxygen per pound of milk solids, whereas only 0.50 pound is required for initial assimilation. Some authorities believe that oxidation "burns up" all the sludge so that none remains for separate disposal (*1*), others claim that suspended solids in the effluent and foaming difficulties experienced at a number of these plants represent residual solids which would settle out in conventional activated sludge systems.

TABLE MK 4.—*Operating results of ¼ aeration (modified activated sludge) type treatment plants*

Type of treatment	Waste volume, gallons per day	Raw waste, B.O.D. pounds per day	Aeration tank, cubic feet per pound 5-day B.O.D. per day	Air supply, cubic feet per pound 5-day B.O.D.	Percent, 5-day B.O.D. removal
Cheese plants					
Aeration, settling sludge return, periodic sludge removal	9,500	179	19	2,000	85
Septic, separate aeration, recirculation through septic tank, settling in 2d septic tank	10,250	213	12	950	96
Aeration, settling sludge return, separate sludge storage	17,200	108	27	1,870	99.4
Receiving station					
Equalization tank aeration, settling sludge return	8,900	17.5	115	5,760	92.7

Temperature is an important variable in the operation of aeration treatment systems. The best results are indicated at about 86° F. At this temperature, endogenous oxidation ("burnup" of the cells) proceeds at about one-tenth the rate of the fermentation of the waste itself. Endogenous oxidation is retarded by lower temperatures and practically ceases at 68° F., and all activity ceases at 50° F. This indicates a need for shelters in cold climates.

In a field study at an individual plant, where B.O.D. reduction was as high as 99 percent, the treatment did not destroy all of the solids produced. It was estimated that approximately 70 percent of the suspended solids in the mixed liquor were eventually destroyed, but 30 percent required special handling. It was observed that all the sludge must be kept in continual circulation and that there be no dead spots where it can accumulate; that the aeration facilities be maintained in continual operation; that the solids of the mixed liquor be kept below 9 percent; and that a waste equalization tank ahead of the aeration tank was desirable to level out fluctuations in strength of waste, volume of waste, and excessive variations in acidity or alkalinity.

Spray Irrigation

Where suitable land is available and cover crop is carefully selected, the disposal of milk waste by spray irrigation is feasible and generally presents the most economical and least troublesome method of treatment. A spray irrigation system consists of either a portable or fixed piping system equipped with irrigation-type sprinkling nozzles. The waste should be screened to prevent clogging of the nozzles, and the discharge pump and wet well should be designed to operate at such a frequency that the wastes in storage will not become septic and that dosage is not so frequent as to flood the disposal area.

The type of cover crop is important. Shallow-rooted grasses are generally recommended and should be well established before starting the spraying operation. With careful operation, properly designed systems can function through the winter in northern climates.

Dosage can be 2,500 to 6,200 gallons per acre per day. The daily rate can be as high as 25,000 gallons, with 3- to 4-day rest periods between applications. Experience indicates that some whey can be discharged with normal cheese-plant wastes without adverse effect upon the system, although excessive doses will harm the grasses and encourage weed growths. Costs range from $4,000 to $5,000 (plus cost of land) for systems handling 15,000 to 20,000 gallons per day, with piping laid on the ground.

Loose loam or gravel soils are preferred. Clay soil is subject to poisoning by the effects of sodium in the waste. Limits of 100-p.p.m. sodium have been suggested. Slightly rolling land, less than 6 percent slope is preferred.

Ridge and Furrow Irrigation

Some dairy plants, particularly in the northern States, have installed ridge and furrow irrigation systems. In this system, wastes are usually discharged in daily dosages. Furrows, 1 to 3 feet deep, and 1 to 3 feet wide, are spaced 3 to 15 feet apart. Control gates regulate the depth of flow to about 6 inches from the ridge tops, and distribution is generally from a main or header ditch to the furrows. On sloping lands furrows can follow the contours. Good grassing is essential to prevent erosion and bank failure.

Ridge and furrow irrigation is generally preferred for strong wastes of low volume, whereas spray irrigation is indicated for larger volumes of normal dairy wastes.

Biological Filters

Biological filters have proven to be adaptable for treating milk waste that is diluted with processing waters. Suspended solids in the wastes may cause difficulties through clogging. Preliminary settling of the waste is desirable; however, some filters are operating successfully without preliminary settling. Studies on

FIGURE 7.—Waste disposal by spray irrigation.

FIGURE 8.—Ridge and furrow irrigation.

high-rate application and recirculation of part of the filter effluent have demonstrated the possibility of producing better treatment efficiencies and reducing the size of the filter required. Although biological filters may not give as high efficiencies as well-operated aeration or activated sludge plants, they are often more economical to operate and do not require as close supervision. Biological filters are capable of producing B.O.D. removals of 75 to 90 percent at loadings of 0.5 to 3.0 pounds B.O.D. per cubic yard.

Two high-rate milk waste treatment plants using dual-type clarifiers and filters effect an overall reduction of suspended solids and B.O.D. of 90 and 95 percent, respectively (2). Results at these two plants are given as follows:

	Plant A	*Plant B*
Flow, G.P.M.	21	31
Recirculation:		
Primary	3.52	1.26
Secondary	1.52	2.23
Total solids (p.p.m.):		
Raw	1,211	1,182
Primary effluent	517	781
Final effluent	461	712
Suspended solids (p.p.m.):		
Raw	363	330
Primary effluent	91	61
Final effluent	36	31
B.O.D. 5-day (p.p.m.):		
Raw	648	533
Primary effluent	96	38
Final effluent	33	20

A high-rate trickling filter pretreating dairy wastes prior to discharge to municipal sewers yields 50 percent B.O.D. removal without settling, with a loading of 3.64 pounds B.O.D. per cubic yard (3).

Activated Sludge

Activated sludge treatment is less elastic and shock-proof than aeration or trickling filters. It has the advantage over trickling filters in that suspended solids or readily flocculated substances in the raw wastes do not interfere with operations. Biochemical reductions of 90 to 95 percent can be obtained with activated-sludge-type plants. For further information, see appended references (4) (5). Since about 1952, interest has turned from activated sludge to aeration (modified activated sludge) plants.

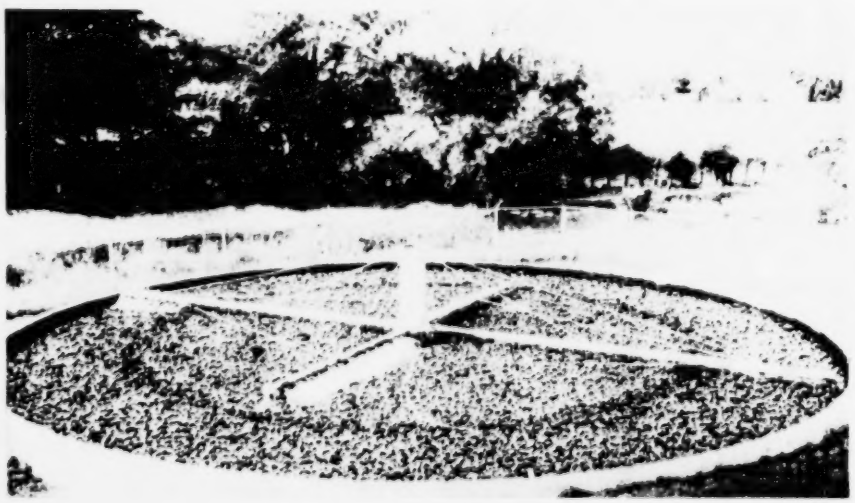


FIGURE 9. Trickling filter.

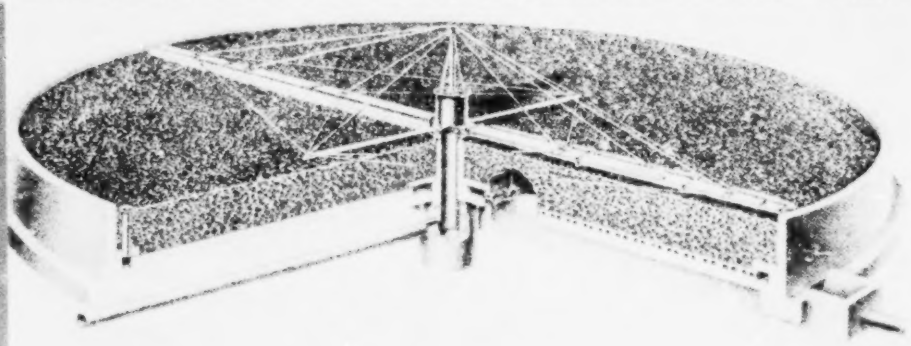


FIGURE 10. Detail of trickling filter construction. The aerobic bacteria living in the slime coating on the stones "digests" the organic matter remaining in the wastes coming from the clarifier.

(1) Porges, N., "Practical Applications of Laboratory Scale Data to Dairy Waste Treatment," Food Technology, 12: 2, pp. 78–80 (1958).

(2) Fischer, A. J., "Duel Clarifier and Filter Structures Simplify High-Rate Treatment Process," CA 41, 7025 f. (1947), R. E. Thompson, Engineering News-Record, 139, 64–66 (1947).

(3) Davy P. S., "Pre-Treatment of Milk Wastes Reduces Treatment Plant Load," Pub. Wks., 83, 1, 56 (1952): Dairy Sci. Abs. 17, 382 (1955).

(4) Montagna S. D., "The Treatment of Dairy Waste," Sewage Works Journal, 12, 108 (1940).

(5) Thayer, Paul M., "New Design for an Activated Sludge Plant To Treat Milk Wastes," p. 171, Proceedings of the Sixth Industrial Waste Conference, Feb. 21–23, 1951, Engineer Bulletin Series No. 76, Purdue University. Designated activated sludge plants by the author, but very similar to aeration-type plants as defined in this publication.

Chemical Oxidation (C.O.D.) Test for Dairy Waste Samples

Test Tube Method

There is need for a test which will supplement the B.O.D. test and give results within a short period of time. Such a test will be of value in the control of waste treatment plants and in the waste prevention program.

Numerous tests have been tried for this purpose, but the one which now shows the most promise is the modified dichromate method given below. This is not meant to supplant the B.O.D. test, but to give more immediate results of a nature comparable to those of the B.O.D. test.

The present modified test has been used for more than 8 years and is a marked improvement over the original Rhame test and its modification by Eldridge, in that replicate portions of oxidizing acid reagent are measured much more accurately and quickly by using a glass syringe with controlled travel of the plunger: the heating time and temperature are more closely controlled by reducing the size of the sample so that ordinary Pyrex test tubes can be used and immersed in an oil bath; and also the controls and a considerable number of samples (six or more) in duplicate can be heated simultaneously. This reduces the spread between duplicates and improves reproducibility between different runs.

Reagents

1. *Dichromate oxidizing agent.* Carefully mix 500 ml. concentrated sulfuric acid, H_2SO_4, sp. gr. 1.84, and 500 ml. 85% orthophosphoric acid, H_3PO_4. Dissolve 2.0 gm. of finely ground C.P. potassium dichromate, $K_2Cr_2O_7$, in the mixture. This will take some time to dissolve but will finally go completely into solution. Before using the dichromate oxidizing agent, any precipitate should be removed by filtering through a layer of asbestos fibers on a glass wool plug in an ordinary glass funnel.

2. *Iodide solution.* Dissolve 50 gm. of sodium iodide, NaI (or 55.4 gm. of potassium iodide, KI), in 200 ml. of distilled water.

3. *Sodium thiosulfate, 0.01 N.* Quantitatively dilute standardized 0.10 *N* or 0.20 *N* sodium thiosulfate with distilled water. (Dilute 25 ml. of 0.2 *N* to 500 ml.) Prepare a fresh 0.01 *N* solution each day. Do not use old 0.01 *N* solutions.

Standard Sodium Thiosulfate

Crystalline sodium thiosulfate, $Na_2S_2O_3 \cdot 5H_2O$, is not a primary standard in exact work. A recommended standardization is that against standard potassium dichromate solution.

For approximately 0.20 *N* thiosulfate solution, dissolve 49.6 gm. C.P. $Na_2S_2O_3 \cdot 5H_2O$ and 1 gm. sodium carbonate, Na_2CO_3, and dilute to 1 liter with freshly boiled, cooled, distilled water. Before use, allow the solution to stand for a day or two away from light (use a dark bottle or dark cupboard).

Where extreme accuracy is desired. Dissolve a slight excess of thiosulfate such as 51.1 gm., add 1 gm. Na_2CO_3, and dilute to 1 liter as above. After storage, standardize against a standard solution of 0.10 *N* potassium dichromate.

Standardization. Place 4 gm. C.P. potassium iodide and 2 gm. C.P. sodium bicarbonate, $NaHCO_3$, in each of 3 or 4 stoppered 600-ml. Erlenmeyer flasks and dissolve in 100 ml. distilled water. Add 5 to 6 ml. concentrated HCl and allow for the evolution of CO_2 gas which displaces oxygen from the flask. Stopper tightly. Pipet exactly 50.0 ml. of standard 0.1 *N* potassium dichromate solution into the flask. Rinse sides of flask with distilled water which layers on the surface. Place in dark for 5 to 6 minutes, dilute to 300–400 ml. with distilled water, and titrate with the sodium thiosulfate solution to be standardized, using starch indicator near endpoint.

Exact normality of thiosulfate =

$$\frac{50.0 \text{ ml. } K_2Cr_2O_7 \times N \text{ of } K_2Cr_2O_7}{\text{ml. } Na_2S_2O_3}$$

Standard $K_2Cr_2O_7$, 0.1 N. Weigh out exactly 4.9035 gm. of C.P. $K_2Cr_2O_7$ (dried at 110° to constant weight) and dilute volumetrically to 1 liter.

4. *Soluble starch solution, 1% solution.* Add 2 gm. starch (potato, arrowroot or soluble) mixed with a little cold water to 200 ml. of boiling distilled water. Allow starch solution to simmer not longer than 5 minutes to prevent hydrolysis of starch. Allow to settle overnight and use only the clear supernatent. Preserve with 0.25 gm. of salicylic acid or 0.8 gm. zinc chloride.

Apparatus

One 250-ml. Pyrex beaker for oxidizing solution
Six 250-ml. Erlenmeyer flasks
One to eight 10-ml. volumetric pipettes for dilution of sample
One to eight 5-ml. volumetric pipettes
Eight 2-ml. volumetric pipettes
One 2-ml. pipette, preferably automatic, for KI solution
One electric hot plate, 660 w. or more at 115 V AC, 3-heat or thermostatic control
Two glass thermometers 0°–250° C.
Eighteen Pyrex test tubes (15 mm. × 150 mm.) with lip
One 50-ml. burette (0.1 ml. div.)
One dropping bottle for starch solution
One 50-ml. graduated cylinder
One oil bath with sufficient (3–6 lbs.) vegetable shortening, such as Crisco, to cover liquid level in sample tubes when rack is immersed. Pyrex beakers, metal coffee pots, and other containers

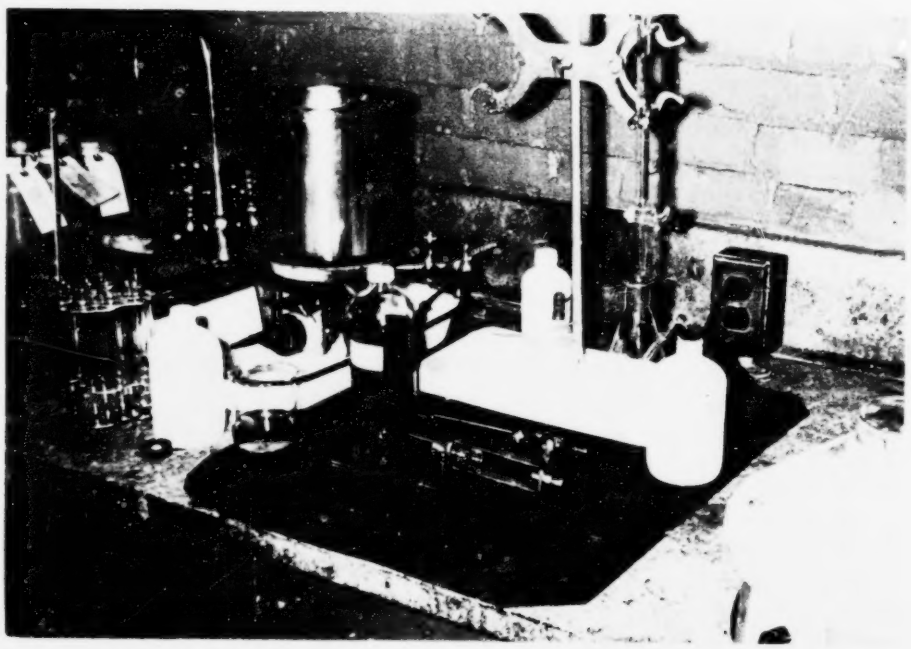


FIGURE 11.—Equipment used in making C.O.D. tests of dairy wastes.

have been used as baths. A straight-sided stainless steel pot with lid ($7^{3}/_{16}$" i.d. x $8^{1}/_{2}$" high) may be used.

One metal test tube rack for immersing tubes in oil bath. Racks holding 14 or 18 tubes may be made to order out of aluminum, copper, or stainless steel.

One 10-ml. Luer syringe, resistance glass

One stainless-steel holder for syringe, with adjustment to limit plunger travel (made to order).

Procedure

1. If practical, preheat the oil bath to about 130° C. before placing on preheated electric plate. Adjust the hot plate control to hold or bring the bath to that temperature which, under prevailing conditions, will heat the tubes of oxidizing solution to 167° C. in 5 minutes.

2. Add exactly 2 ml. distilled water to each of three tubes as blanks and exactly 2 ml. of each waste sample (or suitable dilution) to each of two other tubes (blank and waste samples should be run in duplicate). Use separate volumetric pipettes for each sample. Place a thermometer (about 250° C.) in one of the blank tubes (not to be titrated). Preferably the 2 ml. of sample or suitable dilution should give a thiosulfate titration about 50 percent of that of the blank; titrations from 20 to 80 percent of blank are usually satisfactory.

3. Deliver *exactly* the same volume, about 10 ml., of dichromate solution into each of the test tubes containing either water blank or waste sample. Use a volumetric pipette or preferably a syringe. The dichromate solution should be mixed with the water or the waste sample by swirling. Preferably use a 10 ml. Luer syringe which is held in a frame which limits exactly the throw of the plunger. Make sure that all air bubbles are eliminated when filling the syringe with oxidizing solution from a beaker. Direct the discharge from the syringe along the side of the test tube so as to mix well with the sample.

4. Place the tubes in the rack and the rack in the oil bath and preferably agitate gently during the heating time. Be sure that the rack and outsides of the tubes are free from moisture when placing in the bath.

5. A temperature of 167° C. should be reached in 5 minutes ±1 minute (maximum variation from 4 to 6 minutes). Above this temperature, the mixture starts to decompose as indicated by fumes in the tube above the liquid. (The time required by the tubes to reach 167° C. varies with conditions of heating of the oil bath.)

6. When tubes reach a temperature of 167° C., remove the rack of tubes and drain in air for 4 to 5 minutes only, and then cool immediately to room temperature by placing test tube rack in a container of cold water, running fresh water into it to keep the temperature down. Two to three minutes are usually adequate.

7. Wipe outside of tubes free from oil or water and transfer quantitatively the contents of each tube into a correspondingly marked 250-ml. Erlenmeyer flask using about 40 ml. of distilled water, measured in a graduated cylinder, or estimated from a plastic wash bottle for rinsing out the test tube. Again cool the diluted solation to room temperature with cold water. Use care to prevent contamination of the solutions of sample with oil, etc.

8. Add 2 ml. of sodium iodide (or potassium iodide) solution rapidly to one flask with constant mixing. Use a volumetric pipette or automatic pipette. Titrate immediately with 0.01 *N* thiosulfate, adding 1 ml. of starch indicator solution near the end of the titration when the color becomes a light yellow. The last few milliliters of thiosulfate should be added drop by drop with constant mixing since the endpoint is not sharp and the reaction slow. The endpoint is from dark blue to a pale blue green.

NOTE.—Titrate each flask before adding the iodide to subsequent flask since iodine is volatile and is lost on standing.

Calculations

Chemical oxygen demand as p.p.m. equals (ml. 0.01 *N* thiosulfate in blank less ml. 0.01 *N* thiosulfate in sample) times 80.0 divided by milliliters of original sample.

Miscellaneous Notes

A distilled water control with 10 ml. of freshly prepared oxidizing agent usually requires a titration of about 40 ml. of 0.01 *N* thiosulfate. The oxidizing power gradually decreases with age of the oxidizing agent and may drop 10 percent in 6 to 8 months.

Accurately measured waste samples with little suspended matter and no volatile alcohols, etc., often show a spread of less than 0.3 ml. 0.01 *N* thiosulfate between duplicate tests.

One disadvantage of this C.O.D. test without reflux is that alcohols, aldehydes, and other volatile fermentation products may, and frequently do, evaporate before reacting entirely with the oxidizing agent. The C.O.D. test may greatly underestimate the polluting effect of such wastes high in these volatile organic materials.

Also, suspended particles are completely dissolved and biologically oxidized materials may show further chemical oxygen demand by this test. High chlorides also exert a C.O.D. Some types of organic compounds are only partially oxidized by this test. However, for raw dairy waste the ratio between the 5-day, 20° C. B.O.D. value and the C.O.D. value usually is about 0.68 to 1 or 0.68 for whole milk and skim milk operations. Where whey constitutes a considerable part of the organic matter, the ratio may drop to 0.50 or 0.55. Depending upon the degree of oxidation, samples from dairy waste aeration plants may show a B.O.D.: C.O.D. ratio as low as 0.20 and treated effluents as low as 0.10 or even less with well oxidized waste.

Even with 0.01 *N* thiosulfate, 1 ml. depletion is equivalent to 40 p.p.m. C.O.D. with a 2-ml. undiluted sample. For accurate results this requires glassware that is chemically clean and free from organic matter. For the test tubes and pipettes, use a final cleaning with chromic acid-sulfuric acid cleaning solution followed by a running tapwater rinse and distilled water rinse. Obviously, contamination of the samples during test with milk powder dust, etc., or splattered oil must be avoided.

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